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TESTING OF HAZMATCAD™ DETECTORS AGAINST CHEMICAL WARFARE AGENTS:

SUMMARY REPORT OF EVALUATION PERFORMED
AT U.S. ARMY SOLDIER BIOLOGICAL AND CHEMICAL COMMAND
(SBCCOM)

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This report characterizes the chemical warfare agent (CWA) detection potential of the commercially available 250 mHz HAZMATCAD™. The HAZMATCAD™ instruments were tested against HD, GB, and GA vapors under various conditions. This report is intended to provide the users concerned with CWA detection an overview of HAZMATCAD™'s CWA detection capabilities.			
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PREFACE

The work described in this report was authorized under TSA Project No. 0201T. This work started in November 2000, was completed in June 2002, and includes information contained in an earlier report under the Domestic Preparedness Evaluation Program.

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AGAINST CHEMICAL WARFARE AGENTS:
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1. INTRODUCTION

This testing was performed to evaluate the performance of the HAZMATCAD™ 250 MHz Chemical Agent Detector following design changes made by the manufacturer. Testing was conducted through a test service agreement (TSA) between Microsensor Systems, Incorporated (Bowling Green, KY), and the Applied Chemistry Team (ACT) of Soldier Biological and Chemical Command (SBCCOM). The purpose was to reconfirm some of the previous findings and to identify possible solutions. An earlier HAZMATCAD™ design was tested at SBCCOM as part of the Domestic Preparedness (DP) Program. This report supplements the test report ECBC TR-238, "Domestic Preparedness Program: Testing of HAZMATCAD™ Detectors Against Chemical Warfare Agents: Summary Report."

2. OBJECTIVE

The objective of this report is to demonstrate the capability and general characteristics of the HAZMATCAD™ 250 MHz SAW (Surface Acoustic Wave) instrument in Chemical Warfare (CW) agent vapors detection. The intent is to provide the emergency responders concerned with CW agent detection an overview of the detection capabilities of the instrument.

3. SCOPE

This evaluation is an attempt to characterize the CW agent vapor detection capability of the HAZMATCAD™ SAW sensor based detection instrument. The agents used were limited to tabun (GA), sarin (GB), and mustard (HD). These were chosen as representative CW agents because they are believed to be the most likely threats. Test procedures follow the established DP Detector Test and Evaluation Protocol described in the Phase 1 Test Report.¹ The test concept was as follows:

- a. Determine the minimum detectable level (MDL), the lowest concentration where repeatable detection readings are achieved for each selected CW agent. The current military Joint Services Operational Requirements (JSOR)² for point sampling detectors served as a guide for detection sensitivity objectives.
- b. Investigate the effects of humidity and temperature on instrument performance.

c. Observe the effects of potential interfering vapors upon instrument performance in the laboratory.

4. EQUIPMENT AND TEST PROCEDURES

4.1 System Description

Microsensor Systems, Inc., 62 Corporate Court, Bowling Green, KY 42103; is the manufacturer of the HAZMATCAD™ (<http://www.microsensorsystems.com>). Instrument description and operating procedures originate from the HAZMATCAD™ User's Guide.³ The HAZMATCAD™ employs an array of three 250MHz SAW (Surface Acoustic Wave) sensors in a handheld portable Chemical Agent Detector (CAD) instrument.

HAZMATCAD™ uses a sample pump to collect and concentrate a vapor sample on the pre-concentrator. After sample collection, the trap is heated and the sample is released into the SAW detector array. This sample collection is an important step to significantly improve the instrument resistance to false positives. The pre-concentrator is capable of efficiently trapping the chemical agents of interest and limiting the collection of non-targeted chemicals. The pre-concentrator also provides another key function. It modulates the time of arrival of the chemical agent into the detector array.

The SAW detection is based on the solubility interaction between a chemical agent and the polymer surface. This detection mechanism is reversible and selective. As chemical agents absorb into the sensor polymer surface, the mass of the polymer coating increases. This mass increase causes a frequency change that is proportional to the concentration of the absorbed chemical agents. Using an array of SAW sensors with different polymers provides a multi-pattern sensor response (fingerprint) that is unique to the class of agent. HAZMATCAD™ combines the fingerprint response patterns as well as the temporal characteristics of the agent desorption profile to make an agent type determination. Thus, the HAZMATCAD™ uses four different mechanisms to separate the responses of CW agents from other gases and vapors. These include concentrator sorbent material, thermal desorption profile, selective polymer coatings, and pattern recognition software.

HAZMATCAD™ operates on a 20 second "Fast Mode" or 120 second "High Sensitivity" mode analysis cycle. Therefore, every 20 or 120 seconds, depending on the operational mode, the HAZMATCAD™ reports an updated analysis to the user. The HAZMATCAD™ does not operate like a real time monitor. It is a CWA detector and alarm, (i.e., a go or no go system for the detection of chemical agents). The "High Sensitivity" mode provides additional sensitivity, typically reducing the detection level by 3 to 6 fold when compared to "Fast Mode" performance levels. At the minimum limit of detection, the response alarm time of the HAZMATCAD™ may require 2 to 3 times the analysis cycle time depending on when the agent is encountered. Typical time to alarm is less than 60 seconds in the "Fast Mode". This analysis time variability is dependent on the agent concentration and the pre-concentrator

collection efficiency. At higher threat vapor concentrations the alarm time is typically one cycle or less.

HAZMATCAD™ will produce an alarm (visible and audible) when the preset threshold levels for the CW agent detection algorithm are matched. The alarm threshold signals are typically set 5 to 10 fold higher than the minimum detectable signals from the SAW sensors. The detector simultaneously detects blister and nerve agents. The detection is identified with a corresponding "H" or "G" alarm at three concentration levels ("LOW", "MEDIUM", and "HIGH"). "Low" alarms occur when the SAW signals reach the preset alarm threshold value. "Medium" alarms occur when the SAW signals are 2 times higher than the alarm threshold signal. "High" alarms occur when the SAW signals are 5 times higher than the alarm threshold signal. The Figure is a photograph of the HAZMATCAD™.

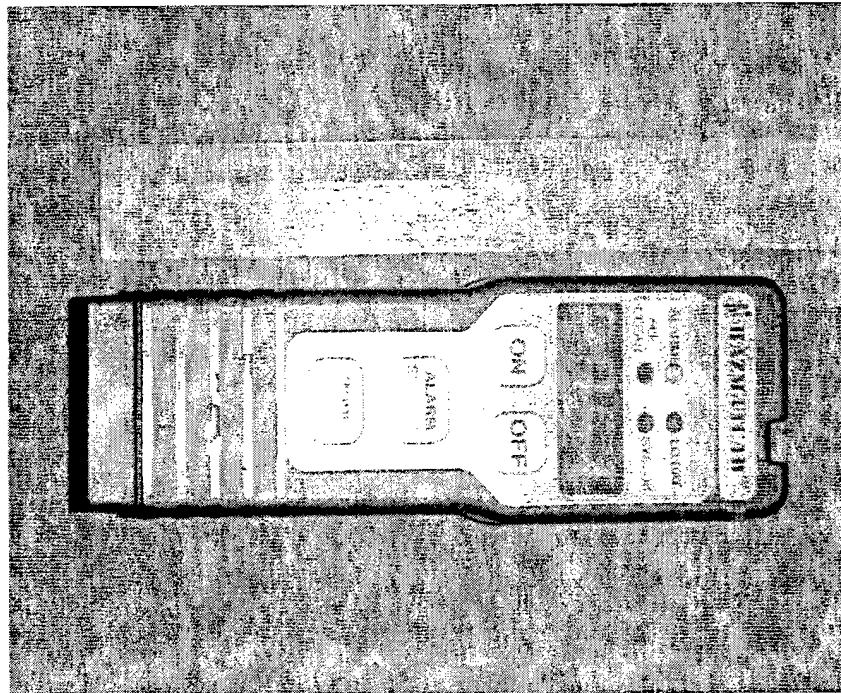


Figure. HAZMATCAD™

The HAZMATCAD™ runs on two commercial SONY NFP-500 lithium-ion (Li-Ion) rechargeable battery packs. Each unit is supplied with an external battery re-charger. The units operate approximately 6-9 hours using the rechargeable battery packs. The battery packs had to be re-charged overnight before each day of testing. The unit weighs 0.64 kg (22 oz) including batteries. After installation of the battery packs, the unit is powered on by pressing the "ON" button. The HAZMATCAD™ is relatively easy to operate and automatically performs a self-diagnostic check, purges itself and begins analyzing for CWA when powered on. According to the Users Manual, the instrument can operate in temperatures from 0 to +40 °C at non-condensing relative humidity (RH) levels of 0 to 95%.

The instrument status is indicated by the LED status display. Two green lights show that the unit is 'ALL CLEAR' and all subsystems are 'SYS OK'. The blinking green 'SYS OK' light indicates proper electrical operation. The steady green 'ALL CLEAR' light indicates that no agents were detected. A yellow 'LOW BATT' indicator light illuminates when the batteries should be charged or replaced. The unit runs continuously and a blinking red 'ALARM' light indicates that an agent has been detected. The alarm status LED flashes at a rate relative to the alarm level. A low concentration threshold level will blink slower than a high level. At the same time, the alphanumeric LED display will flash "H" or "G" for the respective agent class, blister or nerve, and show the relative concentration level.

4.2 Calibration.

No daily instrument calibration is required by the manufacturer to place the HAZMATCAD™ into operation, but a semi-quantitative simulant exposure ("confidence check") is recommended. This confidence check was performed daily during this test. The manufacturer provided a Vapor Simulant Check Source, which is a Teflon vapor diffusion tube that contains DMMP (Dimethyl methyl phosphonate, a G-agent simulant). The Vapor Simulant Check Source allows a total system operational performance check of the instrument.

To perform the confidence check, the HAZMATCAD™ must be in Fast Mode and operating for at least 15 minutes with the appropriate green lights illuminated. Upon exposure to the Vapor Simulant Check Source, a "G" alarm occurs at the end of the measurement cycle to confirm that the instrument is functioning correctly.

4.3 Agent Challenge.

The agent challenges were conducted using the Multi-Purpose Chemical Agent Vapor Generation System⁴ with Chemical Agent Standard Analytical Reference Material (CASARM) grade or highest purity CW agents available. Agent challenge followed successful instrument start up and confidence check. The vapor generator system permits testing of the instrument with humidity and temperature-conditioned air without agent vapor before challenging it with similarly conditioned air containing the CW agent vapor. This is to assure that the temperature and RH conditioned background air does not cause interference with the instrument.

The HAZMATCAD™ inlet is placed under the cup-like sampling port of the vapor generator and exposed to the conditioned air to establish a stable background before agent challenges. Agent challenge begins when the solenoids of the vapor generation system are energized to switch the air streams from conditioned air only to similarly conditioned air containing the agent. The time that the detector was exposed to the agent vapor until it alarmed was recorded as the response time. The agent challenge time was extended to 3-10 min if the detector did not produce an alarm in 2 min to observe its actual response over several additional analysis cycles. This was done to simulate actual application of these instruments. The time required after agent exposures until the instrument stopped alarming was recorded as the recovery time. Each unit was tested three times under each condition.

The instruments were each tested with the agents GA, GB, and HD at several concentration levels at ambient temperatures and 50% RH to determine the MDL with each agent. The effect of humidity on the detectors was also assessed by testing at ambient temperature with <10% and >90% RH. The effects of low temperature were assessed by testing at 0°C for GA, GB, and HD. The high temperatures effects were tested at +40°C for GA, GB, and HD. Temperature extremes were selected based on the manufacturer's stated operating range using agent concentrations that approximated the MDL. Although HD freezes at approximately +15 °C, the calculated HD volatility of 92 mg/m³ at 0 °C easily produces a vapor concentration higher than the 2 mg/m³ JSOR detection criteria allowing the instrument to be evaluated at 0 °C.

4.4 Agent Vapor Quantification.

The generated agent vapor concentrations were analyzed independently and reported in both milligrams per cubic meter (mg/m³) and parts-per-million (ppm) units in the data tables. The vapor concentration was quantified by utilizing the manual sample collection methodology⁵ using the Miniature Continuous Air Monitoring System (MINICAMS[®]) manufactured by O. I. Analytical, Inc. (Birmingham, AL). The MINICAMS[®] is equipped with a flame photometric detector (FPD), and was operated in either phosphorus mode for the GA and GB agents or sulfur mode for HD.

This system normally monitors air by collection through sample lines and subsequently adsorbing the CW agent onto the solid sorbent contained in a glass tube referred to as the pre-concentrator tube (PCT). The PCT is located after the MINICAMS[®] inlet. The concentrated sample was periodically heat desorbed into a gas chromatographic capillary column for subsequent separation, identification, and quantification. For manual sample collection, the PCT is removed from the MINICAMS[®] during the sampling cycle and connected to a measured suction source to draw the vapor sample from the agent generator. The PCT was then re-inserted into the MINICAMS[®] for analysis. This "manual sample collection" methodology eliminated potential loss of sample along the sampling lines and the inlet assembly when the MINICAMS[®] was used as an analytical instrument. The calibration of the MINICAMS[®] was performed daily using the appropriate standards for the agent of interest. The measured mass equivalent (derived from the MINICAMS[®] chromatogram) divided by the total volume (flow rate x time) of the vapor sample drawn through the PCT produces the sample concentration that converts into milligram/cubic meters.

4.5 Laboratory Interference Tests.

The laboratory interference tests were designed to assess the effect on the instruments of vapor exposure from potential interfering substances. The substances were chosen based on the likelihood of their presence during an emergency response by first responders. Additionally, the laboratory interference tests were conducted to assess the CW agent detection capability in the presence of these interferent vapors.

The HAZMATCAD™ units were tested against 1% of the headspace concentrations of diesel fuel, floor wax, AFFF, Spray 9 cleaner, Windex, toluene, and vinegar vapors. The units were also tested against 25 ppm NH₃ (ammonia). If the detector false alarmed at 1% concentration, it was tested at the 0.1% concentration of the substance. A dry air stream carries the headspace vapor of the substance by sweeping it over the liquid in a tube or through the liquid in a bubbler to prepare the interferent gas mixture. Thirty milliliters/minute or 3 ml/min of this vapor saturated air was then diluted to 3 l/min with the conditioned air at ambient temperatures and 50 %RH to produce the 1% or 0.1% concentration of interferent test mixture, respectively.

5. RESULTS AND DISCUSSION

The HAZMATCAD™ units tested and reported in February 2002 under the Domestic Preparedness Program Domestic Preparedness Program “Testing of HAZMATCAD™ Detectors Against Chemical Warfare Agents: Summary Report, February 2002” posted in the website, http://www2.sbccom.army.mil/hld/ip/hazmat_cad_detectors_download.htm, revealed many un-predicted behaviors during the evaluation. Performance of those detectors was erratic at different times throughout that evaluation including inconsistent responses, erratic behaviors, and frequent detector malfunctions. Those operational deficiencies were not observed during this testing. No detector failures were observed.

5.1 Minimum Detectable Levels.

The minimum detectable limit (MDL) for the upgraded HAZMATCAD™ instruments, are shown in Table 1 for each agent at ambient temperatures and 50% RH. The MDL values represent the lowest CW agent concentration that produced three consistent response alarms in three independent trials. Table 1 shows the range of response times observed for the MDL listed in the “Fast Mode”. The MDL concentrations are expressed in milligrams per cubic meter (mg/m³) with equivalent parts per million (ppm) values given in parentheses.

For comparison, the current military JSOR requirements for CW agent sensitivity for point detection alarms, the U.S. Army’s established values for Immediate Danger to Life or Health (IDLH), and the Airborne Exposure Limit (AEL) are also listed in Table 1. Army Regulation (AR) 385-61⁶ is the source for the IDLH and AEL values for GA and GB, and the AEL value for HD. The AR 385-61 does not establish an IDLH for HD due to concerns over carcinogenicity.

In Fast Mode, the units were consistent in their ability to detect GA, GB and HD at levels close to the JSOR or IDLH levels. The MDL of GB was up to 8.5 times higher than the JSOR and IDLH for both units. The HAZMATCAD™ was unable to detect to the AEL values for HD, GA, or GB.

Table 1. Minimum Detectable Level (MDL) and Average Response Time at Ambient Temperatures and 50% RH for the HAZMATCAD™ Units, With Requirements

AGENT and Detector Mode	Concentration in milligrams per cubic meter, mg/m ³ , With parts per million values in parenthesis (ppm) And Response Times				
	Unit A MDL	Unit B MDL	JSOR*	IDLH**	AEL***
HD Fast Mode	1.37 (0.056) in 44-57 sec	1.37 (0.056) in 69-88 sec	2.0 (0.300) in 120 sec	N/A	0.003 (0.0005) up to 8 hr
GA Fast Mode	0.22 (0.032) in 30-34 sec	0.22 (0.032) in 42-47 sec	0.1 (0.015) in 30 sec	0.2 (0.03) up to 30 min	0.0001 (0.000015) up to 8 hr
GB Fast Mode	0.85 (0.14) in 43-46 sec	0.85 (0.14) in 82-89 sec	0.1 (0.017) in 30 sec	0.2 (0.03) up to 30 min	0.0001 (0.000017) up to 8 hr

* Joint Service Operational Requirements for detectors.

** Immediate Danger to Life or Health values from AR 385-61 to determine level of CW protection.

Personnel must wear full ensemble with SCBA for operations or full-face piece respirator for escape.

*** Airborne Exposure Limit values from AR 385-61 to determine masking requirements. Personnel can operate for up to 8 hr unmasked.

5.2 Temperature and Humidity Effects.

The units were tested under manufacturer's specifications for temperature and humidity conditions to assess their responses. Tables 2 through 4 report the respective responses of both units in the "Fast Mode." The detectors were tested at temperature extremes of 0°C and +40°C for HD, GA, and GB.

Temperature extremes appear to degrade the performance the HAZMATCAD™ units. Units would power up on at 0°C and required 15 to 25 minutes to begin analysis. During this period the HAZMATCAD™ would stay in the "Warm Up" mode as indicated on the front panel LED. At 0°C neither unit would alarm to GA in "Fast Mode". The manufacturer representative determined that the SAW Array heater was not reaching its desired operating temperature of 15°C. It was determined that the units would alarm to GA if the temperature array reached 15°C. Microsensor Systems has since re-designed the heater for the SAW array that will provide additional heat to reach the correct operating temperature. The units were able to detect GB and HD at the low operating temperature. Both units required longer recovery times at the colder temperatures.

Table 2. HAZMATCAD™ “Fast Mode” Responses to HD Vapor Concentrations

Temp. °C	%RH	HD Challenge Concentration		Unit A		Unit B	
		mg/m ³	ppm	Alarm Reading	Response Time Range, seconds	Alarm Reading	Response Time Range, seconds
0	0	1.3	0.18	Low H	44-46	Low H	54-101
40	45	2.4	0.34	Low H	34-44	Low H	51-63

Table 3. HAZMATCAD™ “Fast Mode” Responses to GA Vapor Concentrations

Temp. °C	%RH	GA Challenge Concentration		Unit A		Unit B	
		mg/m ³	ppm	Alarm Reading	Response Time Range, seconds	Alarm Reading	Response Time Range, seconds
0	0	0.17	0.03	None*	N/A	None*	N/A
40	45	0.3	0.05	Low G	34-44	Low G	51-63

* Pattern for GA did not match at 0°C, though adequate alarm signal was present.

Table 4. HAZMATCAD™ “Fast Mode” Responses to GB Vapor Concentrations

Temp. °C	%RH	GB Challenge Concentration		Unit A		Unit B	
		mg/m ³	ppm	Alarm Reading	Response Time Range, seconds	Alarm Reading	Response Time Range, seconds
0	0	0.6	0.11	Low G	44-46	Low G	54-101
40	45	2.8	0.51	Low G	34-44	Low G	51-63

High temperature also affected the agent responses of the detectors. Both units A and B alarmed to all the agents in the “Fast Mode”. Operating temperatures of +40°C reduced the collection efficiency of the pre-concentrator, especially the more volatile GB, leading to higher concentration levels required for detection. Humidity changes did not appear to cause adverse effect on the HAZMATCAD™ as evidenced by the detection capability at the high moisture extreme at 45%RH at 40°C.

5.3

Laboratory Interference Tests.

The results of the interference tests are detailed in Table 5. The HAZMATCAD™ performed very well in the false positive testing. Only one false positive was when exposed to the 1% concentration level of Windex Glass Cleaner. This alarm only occurred after 102 seconds of exposure to alarm as "Low G". HAZMATCAD™ did not alarm on Windex exposures at the 0.1% concentration.

Table 5. HAZMATCAD™ Interference Test Summary

Chemical Tested	Unit A 1% Concentration Level	Unit A 0.1% Concentration Level	Unit B 1% Concentration Level	Unit B 0.1% Concentration Level
1% AFFF	No Alarm	Not Tested	No Alarm	Not Tested
Gasoline	No Alarm	Not Tested	No Alarm	Not Tested
JP-8	No Alarm	Not Tested	No Alarm	Not Tested
Toluene	No Alarm	Not Tested	No Alarm	Not Tested
Floor Wax	No Alarm	Not Tested	No Alarm	Not Tested
Spray 9	No Alarm	Not Tested	No Alarm	Not Tested
Windex	Low G	No Alarm	Low G	No Alarm
Diesel	No Alarm	Not Tested	No Alarm	Not Tested
Bleach	No Alarm	Not Tested	No Alarm	Not Tested
Ammonia	No Alarm	Not Tested	No Alarm	Not Tested

6.

CONCLUSIONS

The improved HAZMATCAD™ instruments performed better than those evaluated in the testing conducted at the U.S. Army Soldier Biological and Chemical Command (SBCCOM) under the Domestic Preparedness Detector Evaluation Program. Most of the observed operational deficiencies, such as inconsistent response, erratic behaviors, and frequent malfunctions were overcome. They operated within the manufacturers specifications and experienced no operational reliability issues. HAZMATCAD™ was able to detect GA, GB and HD at detection levels that were above the JSOR or IDLH levels for GA and GB but below the JSOR or IDLH levels for HD. Humidity did not appear to cause adverse effects on the performance of the HAZMATCAD™.

The detection capabilities of the HAZMATCAD™ were degraded at the high and low temperature extremes. In cold temperatures, the units were unable to detect GA at 0°C due to the inability of the sensor array heater to maintain the required operating temperature for a

correct fingerprint pattern match. Microsensor Systems, Inc. (Bowling Green, KY) has stated that they plan to redesign the sensor array heater to improve the SAW array temperature control at low temperatures. At high temperatures HAZMATCAD™ alarmed to all of the agents at the concentration values that were 0.5 to 2 times higher than the room temperature concentrations.

HAZMATCAD™ only false alarmed to one of the ten potential interference vapors. That was at the 1% headspace concentration level of Windex Glass Cleaner at ambient temperature. Windex vapor at the .1% level did not cause the HAZMATCAD™ to false alarm. HAZMATCAD™ was not tested with CW agent in the presence of interferent chemicals during the TSA.

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